PCT/EP2003/008944

## EVALUATION (D-SPACINGS, RELATIVE INTENSITIES) OF POWDER X-RAY DIFFRACTION PATTERNS

#### **Equipment** used

X-Ray Powder Diffractometer D-8 (AXS-BRUKER) theta-theta-goniometer, sample changer target: Copper,  $K\alpha 1+K\alpha 2$   $\lambda=1.5406$  Å parallel beam optics (receiving soller-slit: 0.07 mm) Scintillation counter, standard sample holders

### Samples and Data-Collection

### Table / Figure

- 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid- **phosphate** data collection: 40kV, 40 mA, 2-40° θ/2θ, 0.01 steps, 2 seconds
- 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid- hydrochloride data collection: 40kV, 40 mA, 2-40° θ/2θ, 0.01 steps, 2 seconds
- 3) 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid- tosylate data collection: 40kV, 40 mA, 2-40° θ/2θ, 0.01 steps, 2 seconds
- 4) 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid- hydrogensulfate data collection: 40kV, 40 mA, 2-40° θ/2θ, 0.01 steps, 2 seconds
- 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid- mesylate
   data collection: 40kV, 40 mA, 2-40° θ/2θ, 0.01 steps, 2 seconds
- 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid- sulfate
   data collection: 40kV, 40 mA, 2-40° θ/2θ, 0.01 steps, 2 seconds

#### external d-spacing standards:

- 1) NIST SRM 640A (Silicon Powder)
- 2) NIST SRM 675 (synth. Fluorophlogopite) data collection: 40kV, 40mA, 2 50° 6/26, 0.01 steps, 2 seconds

#### **Software**

DIFFRAC-Plus and TOPAS (AXS-BRUKER)

# 10/524397

### External d-spacing and relative intensity evaluation of the powder diffractometer (D8 - AXS-BRUKER) with NIST standards:

SRM 640A:	28.443° <sub>expected</sub> 47.304° <sub>expected</sub> Rel.Intensity 100 <sub>expected</sub> Rel.Intensity 55 <sub>expected</sub>	28.446° measured 47.308° measured Rel. Intensity 100 measured Rel. Intensity 55 measured
SRM 675:	8.853° expected 17.759° expected 26.774° expected 35.962° expected 45.397° expected	8.849° measured 17.754° measured 26.778° measured 35.962° measured 45.397° measured
	Rel.Intensity 81 <sub>expected</sub> Rel.Intensity 5 <sub>expected</sub> Rel.Intensity 100 <sub>expected</sub> Rel.Intensity 7 <sub>expected</sub> Rel.Intensity 28 <sub>expected</sub>	Rel. Intensity 80 measured Rel. Intensity 100 measured Rel. Intensity 6 measured Rel. Intensity 27 measured Rel. Intensity 27 measured

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## POWDER PATTERN (D-I-LIST)

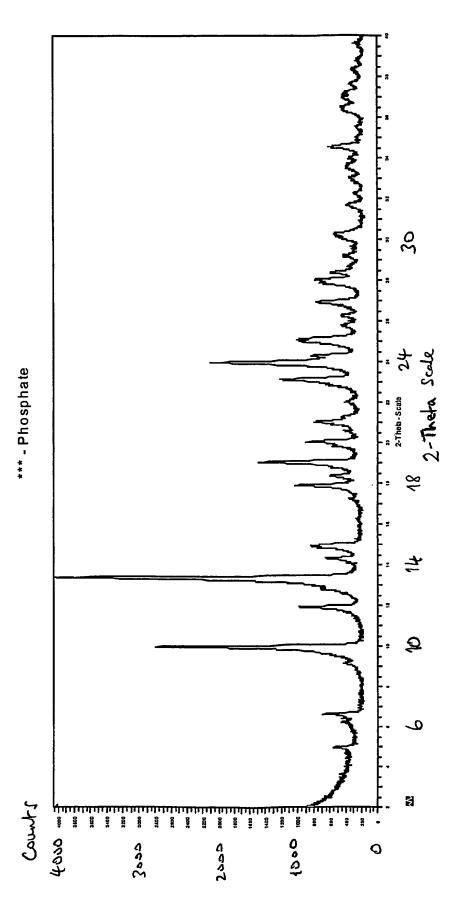
7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid phosphate

Table 1

17.813	l value	Angle	Intensity
14.200       6.22       6         13.342       6.62       13         9.696       9.11       7         8.860       9.98       68         7.422       11.92       21         6.923       12.78       14         6.636       13.33       100         6.195       14.29       13         5.940       14.90       18         5.459       16.23       3         5.148       17.21       5         4.963       17.86       23         4.823       18.38       11         4.661       19.03       34         4.474       19.83       11         4.420       20.07       19         4.309       20.60       7         4.221       21.03       17         4.173       21.27       8         4.035       22.01       6         3.844       23.12       27         3.663       24.97       21         3.663       24.97       21         3.663       24.97       21         3.533       25.19       22         3.446       25.83       8			(Rel. Int)
13.342       6.62       13         9.698       9.11       7         8.860       9.98       68         7.422       11.92       21         6.923       12.78       14         6.636       13.33       100         6.195       14.29       13         5.940       14.90       18         5.459       16.23       3         5.148       17.21       5         4.963       17.86       23         4.823       18.38       11         4.661       19.03       34         4.474       19.83       11         4.420       20.07       19         4.309       20.60       7         4.173       21.27       8         4.035       22.01       6         3.890       22.85       13         3.661       24.29       17         3.663       24.97       21         3.533       25.19       22         3.303       26.97       16         3.196       27.89       15         3.196       27.89       15         3.142       28.39       11 <td>17.813</td> <td>4.96</td> <td>7</td>	17.813	4.96	7
9.696 9.11 7 8.860 9.98 68 7.422 11.92 21 6.923 12.78 14 6.636 13.33 100 6.195 14.29 13 6.940 14.90 18 5.459 16.23 3 5.148 17.21 5 4.963 17.86 23 4.823 18.38 11 4.661 19.03 34 4.474 19.83 11 4.420 20.07 19 4.309 20.60 7 4.309 20.60 7 4.309 20.60 7 4.173 21.27 8 4.035 22.01 6 3.890 22.85 13 3.844 23.12 27 3.707 23.98 50 3.844 23.12 27 3.707 23.98 50 3.661 24.29 17 3.663 24.97 21 3.663 24.97 21 3.663 25.19 22 3.446 25.83 8 3.387 26.29 8 3.303 26.97 16 3.196 27.89 15 3.176 28.08 16 3.142 28.39 11 3.105 28.73 55 3.051 29.25 7 3.014 29.62 4 2.948 30.30 10 2.817 31.74 6 2.671 33.53 6 2.671 33.53 6 2.671 33.53 6 2.671 33.53 6 2.671 33.53 6 2.671 33.53 6	14.200	8.22	6
8.860       9.98       68         7.422       11.92       21         6.923       12.78       14         6.636       13.33       100         6.195       14.29       13         5.940       14.90       18         5.945       16.23       3         5.148       17.21       5         4.963       17.86       23         4.823       18.38       11         4.661       19.03       34         4.474       19.83       11         4.420       20.07       19         4.309       20.60       7         4.221       21.03       17         4.173       21.27       8         4.035       22.01       6         3.890       22.85       13         3.844       23.12       27         3.663       24.97       21         3.663       24.97       21         3.633       25.19       22         3.303       26.97       16         3.196       27.89       15         3.175       28.08       16         3.142       28.39       11<	13.342	6.62	13
7.422 11.92 21 6.923 12.78 14 6.636 13.33 100 6.195 14.29 13 5.940 14.90 18 5.459 16.23 3 5.148 17.21 5 4.963 17.86 23 4.823 18.38 11 4.661 19.03 34 4.474 19.83 11 4.420 20.07 19 4.309 20.60 7 4.309 20.60 7 4.221 21.03 17 4.173 21.27 8 4.035 22.01 6 3.890 22.85 13 3.844 23.12 27 3.707 23.98 50 3.844 23.12 27 3.707 23.98 50 3.661 24.29 17 3.563 24.97 21 3.563 24.97 21 3.563 25.19 22 3.303 26.97 16 3.196 27.89 15 3.175 28.08 16 3.142 28.39 11 3.105 28.73 5 3.051 29.25 7 3.014 29.62 4 2.948 30.30 10 2.817 31.74 6 2.770 32.30 3 2.709 33.04 5 2.671 33.53 66 2.651 33.74 6			-
6.923       12.78       14         6.636       13.33       100         6.195       14.29       13         5.940       14.90       18         5.459       16.23       3         5.148       17.21       5         4.963       17.86       23         4.823       18.38       11         4.661       19.03       34         4.474       19.83       11         4.420       20.07       19         4.308       20.60       7         4.221       21.03       17         4.173       21.27       8         4.035       22.01       6         3.890       22.86       13         3.844       23.12       27         3.661       24.29       17         3.633       25.19       22         3.446       25.83       8         3.397       26.29       8         3.303       26.97       16         3.196       27.89       15         3.175       28.08       16         3.142       28.39       11         3.051       29.25       7 <td></td> <td></td> <td></td>			
6.636       13.33       100         6.195       14.29       13         5.940       14.90       18         5.459       16.23       3         5.148       17.21       5         4.963       17.86       23         4.823       18.38       11         4.661       19.03       34         4.474       19.83       11         4.420       20.07       19         4.309       20.60       7         4.309       20.60       7         4.035       22.01       6         3.890       22.86       13         3.844       23.12       27         3.631       24.29       17         3.533       25.19       22         3.466       25.83       8         3.387       26.29       8         3.303       26.97       16         3.196       27.89       15         3.175       28.08       16         3.142       28.73       5         3.051       29.25       7         3.014       29.62       4         2.948       30.30       10			
6.195       14.29       13         5.940       14.90       18         5.459       16.23       3         5.148       17.21       5         4.963       17.86       23         4.823       18.38       11         4.661       19.03       34         4.474       19.83       11         4.420       20.07       19         4.309       20.60       7         4.309       20.60       7         4.035       22.01       6         3.890       22.85       13         3.844       23.12       27         3.661       24.29       17         3.533       25.19       22         3.533       25.19       22         3.446       25.83       8         3.387       26.29       8         3.196       27.89       15         3.176       28.08       16         3.142       28.73       5         3.051       29.25       7         3.051       29.25       7         3.051       29.25       7         3.074       29.62       4			
5.940       14.90       18         5.459       16.23       3         5.148       17.21       5         4.963       17.86       23         4.823       18.38       11         4.661       19.03       34         4.474       19.83       11         4.420       20.07       19         4.309       20.60       7         4.221       21.03       17         4.173       21.27       8         4.035       22.01       6         3.890       22.85       13         3.844       23.12       27         3.707       23.98       50         3.661       24.29       17         3.533       25.19       22         3.446       25.83       8         3.387       26.29       8         3.303       26.97       16         3.196       27.89       15         3.175       28.08       16         3.142       28.39       11         3.051       29.25       7         3.014       29.62       4         2.948       30.30       10			
5.469       16.23       3         5.148       17.21       5         4.963       17.86       23         4.823       18.38       11         4.661       19.03       34         4.474       19.83       11         4.420       20.07       19         4.309       20.60       7         4.221       21.03       17         4.173       21.27       8         4.035       22.01       6         3.890       22.85       13         3.844       23.12       27         3.707       23.98       50         3.661       24.29       17         3.533       25.19       22         3.446       25.83       8         3.387       26.29       8         3.196       27.89       15         3.175       28.08       16         3.142       28.39       11         3.051       29.25       7         3.014       29.82       4         2.948       30.30       10         2.817       31.74       6         2.709       33.04       5			
5.148       17.21       6         4.963       17.86       23         4.823       18.38       11         4.661       19.03       34         4.474       19.83       11         4.420       20.07       19         4.309       20.60       7         4.221       21.03       17         4.173       21.27       8         4.035       22.01       6         3.890       22.85       13         3.844       23.12       27         3.707       23.98       50         3.661       24.29       17         3.533       25.19       22         3.446       25.83       8         3.387       26.29       8         3.196       27.89       15         3.175       28.08       16         3.142       28.39       11         3.051       29.25       7         3.051       29.25       7         3.051       29.25       7         3.051       29.25       7         3.051       29.25       7         3.051       29.26       7			
4.963       17.86       23         4.823       18.38       11         4.661       19.03       34         4.474       19.83       11         4.420       20.07       19         4.309       20.60       7         4.221       21.03       17         4.173       21.27       8         4.035       22.01       6         3.890       22.85       13         3.844       23.12       27         3.707       23.98       50         3.661       24.29       17         3.533       25.19       22         3.533       25.19       22         3.466       25.83       8         3.397       26.29       8         3.303       26.97       16         3.196       27.89       15         3.176       28.08       16         3.142       28.39       11         3.051       29.25       7         3.014       29.62       4         2.948       30.30       10         2.817       31.74       2         2.948       30.30       3			
4.823       18.38       11         4.661       19.03       34         4.474       19.83       11         4.420       20.07       19         4.309       20.60       7         4.221       21.03       17         4.173       21.27       8         4.035       22.01       6         3.890       22.85       13         3.844       23.12       27         3.661       24.29       17         3.633       25.19       22         3.446       25.83       8         3.387       26.29       8         3.303       26.97       16         3.196       27.89       15         3.175       28.08       16         3.142       28.39       11         3.051       29.25       7         3.014       29.62       4         2.948       30.30       10         2.817       31.74       6         2.709       33.04       5         2.671       33.53       6         2.654       33.74       6			_
4.661       19.03       34         4.474       19.83       11         4.420       20.07       19         4.309       20.60       7         4.221       21.03       17         4.173       21.27       8         4.035       22.01       6         3.890       22.85       13         3.844       23.12       27         3.707       23.98       50         3.661       24.29       17         3.683       24.97       21         3.633       25.19       22         3.446       25.83       8         3.387       26.29       8         3.303       28.97       16         3.196       27.89       15         3.175       28.08       16         3.142       28.39       11         3.051       29.25       7         3.014       29.62       4         2.948       30.30       10         2.817       31.74       6         2.871       33.63       6         2.871       33.53       6         2.671       33.53       6			
4.420       20.07       19         4.309       20.60       7         4.221       21.03       17         4.173       21.27       8         4.035       22.01       6         3.890       22.85       13         3.844       23.12       27         3.707       23.98       50         3.661       24.29       17         3.563       24.97       21         3.633       25.19       22         3.446       25.83       8         3.397       26.29       8         3.196       27.89       15         3.176       28.08       16         3.142       28.39       11         3.051       29.25       7         3.014       29.62       4         2.948       30.30       10         2.817       31.74       6         2.709       33.04       5         2.671       33.53       6         2.654       33.74       6			34
4.308       20.60       7         4.221       21.03       17         4.173       21.27       8         4.035       22.01       6         3.890       22.85       13         3.844       23.12       27         3.707       23.98       50         3.661       24.29       17         3.633       24.97       21         3.633       25.19       22         3.446       25.83       8         3.387       26.29       8         3.303       26.97       16         3.196       27.89       15         3.176       28.08       16         3.142       28.39       11         3.105       28.73       5         3.014       29.62       4         2.948       30.30       10         2.817       31.74       6         2.709       33.04       5         2.671       33.53       6         2.654       33.74       6	4.474	19.83	11
4.221       21.03       17         4.173       21.27       8         4.035       22.01       6         3.890       22.85       13         3.844       23.12       27         3.707       23.98       50         3.661       24.29       17         3.563       24.97       21         3.533       25.19       22         3.446       25.83       8         3.387       26.29       8         3.303       26.97       16         3.196       27.89       15         3.175       28.08       16         3.142       28.39       11         3.051       29.25       7         3.051       29.25       7         3.014       29.62       4         2.948       30.30       10         2.817       31.74       6         2.709       33.04       5         2.671       33.53       6         2.654       33.74       6	4.420	20.07	19
4.173       21.27       8         4.035       22.01       6         3.890       22.85       13         3.844       23.12       27         3.707       23.98       50         3.661       24.29       17         3.563       24.97       21         3.633       25.19       22         3.446       25.83       8         3.387       26.29       8         3.303       26.97       16         3.196       27.89       15         3.175       28.08       16         3.142       28.39       11         3.051       29.25       7         3.051       29.25       7         3.014       29.62       4         2.948       30.30       10         2.817       31.74       6         2.709       33.04       5         2.671       33.53       6         2.654       33.74       6	4.309	20.60	7
4.035       22.01       6         3.890       22.85       13         3.844       23.12       27         3.707       23.98       50         3.661       24.29       17         3.563       24.97       21         3.533       25.19       22         3.446       25.83       8         3.387       26.29       8         3.303       26.97       16         3.196       27.89       15         3.175       28.08       16         3.142       28.39       11         3.051       29.25       7         3.014       29.62       4         2.948       30.30       10         2.817       31.74       6         2.709       33.04       5         2.671       33.53       6         2.654       33.74       6	4.221	21.03	17
3.890       22.85       13         3.844       23.12       27         3.707       23.98       50         3.661       24.29       17         3.563       24.97       21         3.533       25.19       22         3.446       25.83       8         3.387       26.29       8         3.303       26.97       16         3.196       27.89       15         3.175       28.08       16         3.142       28.39       11         3.051       29.25       7         3.051       29.25       7         3.014       29.62       4         2.948       30.30       10         2.817       31.74       6         2.709       33.04       5         2.671       33.53       6         2.654       33.74       6			8
3.844     23.12     27       3.707     23.98     50       3.661     24.29     17       3.563     24.97     21       3.533     25.19     22       3.446     25.83     8       3.387     26.29     8       3.303     26.97     16       3.196     27.89     15       3.175     28.08     16       3.142     28.39     11       3.051     28.73     5       3.051     29.25     7       3.014     29.62     4       2.948     30.30     10       2.817     31.74     6       2.709     33.04     5       2.671     33.53     6       2.654     33.74     6			_
3.707     23.98     50       3.661     24.29     17       3.563     24.97     21       3.533     25.19     22       3.446     25.83     8       3.387     26.29     8       3.303     26.97     16       3.196     27.89     15       3.175     28.08     16       3.142     28.39     11       3.051     29.25     7       3.051     29.25     7       3.014     29.62     4       2.948     30.30     10       2.817     31.74     6       2.709     33.04     5       2.671     33.53     6       2.654     33.74     6			
3.661     24.29     17       3.563     24.97     21       3.533     25.19     22       3.446     25.83     8       3.387     26.29     8       3.303     26.97     16       3.196     27.89     15       3.175     28.08     16       3.142     28.39     11       3.051     28.73     5       3.051     29.25     7       3.014     29.62     4       2.948     30.30     10       2.817     31.74     6       2.709     33.04     5       2.671     33.53     6       2.654     33.74     6			
3.563     24.97     21       3.533     25.19     22       3.446     25.83     8       3.387     26.29     8       3.303     28.97     16       3.196     27.89     15       3.176     28.08     16       3.142     28.39     11       3.051     29.25     7       3.014     29.62     4       2.948     30.30     10       2.817     31.74     6       2.770     32.30     3       2.709     33.04     5       2.671     33.53     6       2.654     33.74     6			
3.533     25.19     22       3.446     25.83     8       3.387     26.29     8       3.303     26.97     16       3.196     27.89     15       3.175     28.08     16       3.142     28.39     11       3.051     28.73     5       3.051     29.25     7       3.014     29.62     4       2.948     30.30     10       2.817     31.74     6       2.709     33.04     5       2.671     33.63     6       2.854     33.74     6			
3.446       25.83       8         3.387       26.29       8         3.303       26.97       16         3.196       27.89       15         3.175       28.08       16         3.142       28.39       11         3.051       29.25       7         3.051       29.25       7         3.014       29.62       4         2.948       30.30       10         2.817       31.74       6         2.709       33.04       5         2.671       33.53       6         2.854       33.74       6			
3.387     26.29     8       3.303     26.97     16       3.196     27.89     15       3.176     28.08     16       3.142     28.39     11       3.051     29.25     7       3.014     29.62     4       2.948     30.30     10       2.817     31.74     6       2.709     33.04     5       2.671     33.53     6       2.654     33.74     6			-
3.196     27.89     15       3.176     28.08     16       3.142     28.39     11       3.051     29.25     7       3.014     29.62     4       2.948     30.30     10       2.817     31.74     6       2.770     32.30     3       2.709     33.04     5       2.671     33.53     6       2.654     33.74     6			8
3.175 28.08 16 3.142 28.39 11 3.105 28.73 5 3.051 29.25 7 3.014 29.62 4 2.948 30.30 10 2.817 31.74 6 2.770 32.30 3 2.709 33.04 5 2.671 33.53 6 2.654 33.74 6			16
3.142     28.39     11       3.105     28.73     5       3.051     29.25     7       3.014     29.62     4       2.948     30.30     10       2.817     31.74     6       2.770     32.30     3       2.709     33.04     5       2.671     33.53     6       2.654     33.74     6	3.196	27.89	15
3.105     28.73     5       3.051     29.25     7       3.014     29.62     4       2.948     30.30     10       2.817     31.74     6       2.770     32.30     3       2.709     33.04     5       2.671     33.53     6       2.654     33.74     6	3.175	28.08	16
3.051     29.25     7       3.014     29.62     4       2.948     30.30     10       2.817     31.74     6       2.770     32.30     3       2.709     33.04     5       2.671     33.53     6       2.654     33.74     6	3.142		11
3.014 28.62 4 2.948 30.30 10 2.817 31.74 6 2.770 32.30 3 2.709 33.04 5 2.671 33.53 6 2.654 33.74 6			5
2.948     30.30     10       2.817     31.74     6       2.770     32.30     3       2.709     33.04     5       2.671     33.53     6       2.854     33.74     6			7
2.817     31.74     6       2.770     32.30     3       2.709     33.04     5       2.671     33.53     6       2.854     33.74     6			-
2.770     32.30     3       2.709     33.04     5       2.671     33.53     6       2.854     33.74     6			
2.709     33.04     5       2.671     33.63     6       2.654     33.74     6			_
2.671 33.53 6 2.654 33.74 6			_
2.654 33.74 6			_
	2.634	33.74 34.05	5
			11
			3
2.481 36.48 8		36.48	8
2.431 36.95 7	2.431		7
		37.28	7
	2.387		5
		38.39	4
2.316 38.85 3 2.273 39.62 3	2.343		_

POWDER PATTERN (DIFFRACTOGRAMM) Figure 1

7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid -phosphate



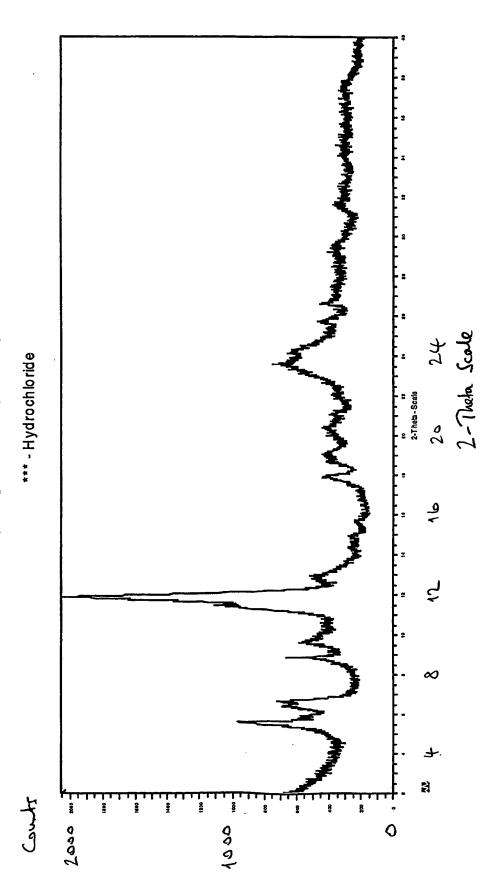
POWDER PATTERN (D-I-LIST)
7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]3-vinyl-3-cephem-4-carboxylic acid hydrochloride

Table 2

HYDROCHLORIDE	2	
d value		Intensity
(Angatrom)	(°CuKok)	(Rel. Int)
15.934	5.54	36
15.176	5.82	16
13.791	6.40	23
13.281	6.65	25
9.965	8.87	24
9.171	9.64	20
7.720	11.45	48
7.460	11.85	100
6.825	12.98	17
6.129	14.44	6
5.940	14.90	6
5.661	15.64	5
4.963	17.86	14
4.744	18.69	13
4.648	19.09	13
4.406	20.14	11
4.328	20.51	13
4.197	21.15	10
4.040	21.98	8
3.760	23.65	27
3.670	24.23	22
3.454	25.77	11
3.348	26.60	11
3.238	27.52	7
3.057	29.19	7
3.017	29.59	7
2.830	31.59	7
2.752	32.51	6
2.631	34.05	6
2.404	37.38	7

POWDER PATTERN (DIFFRACTOGRAMM) Figure 2

7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid hydrochloride



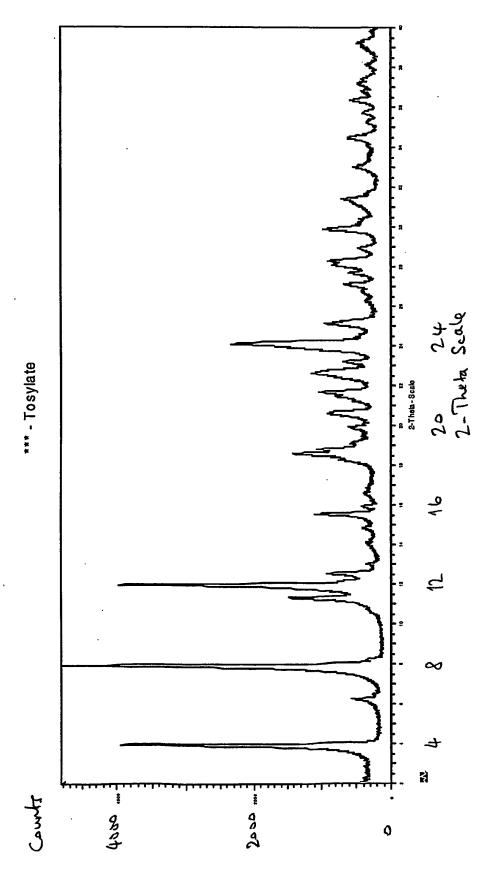
POWDER PATTERN (D-I-LIST)
7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]3-vinyl-3-cephem-4-carboxylic acid tosylate

Table 3

dvalue (Angle (* Cu ka) (Rel. Int)	TOSYLATE		
22.579         3.91         80           14.130         8.25         9           13.660         6.47         3           11.165         7.91         100           10.747         8.22         5           8.322         10.62         4           7.825         11.30         28           7.403         11.95         81           7.041         12.56         16           6.777         13.05         4           6.289         14.07         4           6.977         14.81         6           6.955         15.55         19           6.553         15.95         5           5.328         16.63         4           4.847         18.29         11           4.775         18.57         26           4.714         18.81         18           4.831         19.15         8           4.309         20.80         14           4.143         21.43         12           4.097         21.88         17           3.928         22.63         19           3.824         23.25         10	d value		
14.130	(Angatrom)	(°CuKos)	(Rel. Int.)
13.660			
11.165			<del>-</del>
10.747			
7.825 11.30 28 7.403 11.95 81 7.041 12.56 10 6.777 13.05 4 6.289 14.07 4 6.977 14.81 6 6.977 14.81 6 6.695 15.55 19 6.553 15.95 5 5.328 16.63 4 4.847 18.29 11 4.775 18.57 26 4.714 18.81 18 4.631 19.15 8 4.460 19.89 8 4.309 20.60 14 4.143 21.43 12 4.097 21.68 17 3.928 22.63 19 3.824 23.25 10 3.717 23.92 28 3.689 24.11 45 3.642 25.12 15 3.383 26.32 3 3.329 26.76 4 3.289 27.09 9 3.213 27.74 8 3.176 28.08 13 3.146 28.38 15 3.086 28.91 6 2.989 29.86 16 2.957 30.20 6 2.938 31.48 8 2.844 31.43 11 2.822 31.68 4 2.714 32.97 7 2.671 33.53 3 2.628 34.08 4 2.714 32.97 7 2.671 33.53 3 2.628 34.08 4 2.599 34.48 9 2.542 35.28 3 2.510 36.76 5 2.471 36.33 8 2.439 38.82 5 2.417 37.16 6 2.471 36.33 8 2.439 38.82 5 2.417 37.16 6 2.400 37.44 5 2.381 38.09 4 2.305 39.04 5 2.293 39.26 6	10.747	8.22	5
7.403			•
7.041 12.56 10 6.777 13.05 4 6.289 14.07 4 6.977 14.81 6 5.695 15.55 19 5.5653 15.95 5 5.328 16.63 4 4.847 18.29 11 4.775 18.57 26 4.714 18.81 18 4.631 19.15 8 4.460 19.89 8 4.309 20.60 14 4.143 21.43 12 4.097 21.68 17 3.926 22.63 19 3.824 23.25 10 3.717 23.92 28 3.889 24.11 45 3.542 25.12 15 3.383 28.32 3 3.329 26.76 4 3.289 27.09 9 3.213 27.74 8 3.175 28.08 13 3.146 28.36 15 3.086 28.91 6 2.957 30.20 6 2.938 30.42 5 2.868 31.16 8 2.944 31.43 11 2.822 31.68 4 2.714 32.97 7 2.671 33.53 3 2.628 34.08 4 2.599 34.48 9 2.542 35.28 3 2.510 35.76 5 2.471 36.33 8 2.439 36.82 5 2.417 37.16 6 2.400 37.44 5 2.305 39.04 5 2.293 39.26 6			
6.289       14.07       4         6.977       14.81       5         5.695       15.55       19         5.553       15.95       5         5.328       16.63       4         4.847       18.29       11         4.775       18.57       26         4.714       18.81       18         4.631       19.15       8         4.460       19.89       8         4.309       20.60       14         4.143       21.43       12         4.097       21.68       17         3.928       22.63       19         3.824       23.25       10         3.717       23.92       28         3.889       24.11       45         3.542       25.12       15         3.383       26.32       3         3.289       27.09       9         3.213       27.74       8         3.146       28.36       15         3.086       28.91       6         2.936       30.42       6         2.936       30.42       6         2.936       34.48       9	7.041		
6.977       14.81       6         5.695       15.55       19         5.553       15.95       5         5.328       16.63       4         4.847       18.29       11         4.775       18.57       20         4.714       18.81       18         4.631       19.15       8         4.460       19.89       8         4.309       20.60       14         4.143       21.43       12         4.097       21.68       17         3.926       22.63       19         3.824       23.25       10         3.717       23.92       28         3.689       24.11       45         3.642       25.12       15         3.393       26.32       3         3.329       26.76       4         3.289       27.09       9         3.213       27.74       8         3.176       28.08       13         3.145       28.36       15         3.080       28.91       6         2.938       30.42       6         2.938       31.16       8			•
5.695       15.55       19         5.553       15.95       5         5.328       18.63       4         4.847       18.29       11         4.775       18.57       26         4.714       18.81       18         4.631       19.15       8         4.309       20.60       14         4.143       21.43       12         4.097       21.68       17         3.926       22.63       19         3.824       23.25       10         3.717       23.92       28         3.689       24.11       45         3.542       25.12       15         3.383       26.32       3         3.329       26.76       4         3.289       27.09       9         3.213       27.74       8         3.176       28.08       13         3.145       28.38       15         3.086       28.91       6         2.989       29.86       16         2.957       30.20       6         2.888       31.16       8         2.844       31.43       11	i e		•
5.328       10.63       4         4.847       18.29       11         4.775       18.57       26         4.714       18.81       18         4.631       19.15       8         4.460       19.89       8         4.309       20.60       14         4.143       21.43       12         4.097       21.68       17         3.926       22.63       19         3.824       23.25       10         3.717       23.92       28         3.689       24.11       45         3.542       25.12       15         3.383       26.32       3         3.299       27.09       9         3.213       27.74       8         3.175       28.08       13         3.146       28.36       15         3.086       28.91       6         2.957       30.20       6         2.938       30.42       5         2.988       31.16       8         2.944       31.43       11         2.822       31.68       4         2.714       32.97       7			
4.847       18.29       11         4.775       18.57       26         4.714       18.81       18         4.631       19.15       8         4.460       19.89       8         4.309       20.60       14         4.143       21.43       12         4.097       21.68       17         3.926       22.63       19         3.824       23.25       10         3.717       23.92       28         3.689       24.11       45         3.542       25.12       15         3.383       26.32       3         3.329       26.76       4         3.289       27.09       9         3.213       27.74       8         3.175       28.08       13         3.145       28.36       15         3.086       28.91       6         2.989       29.86       16         2.936       30.42       5         2.936       31.16       8         2.844       31.43       11         2.822       31.88       4         2.714       32.97       7			
4.775       18.57       26         4.714       18.81       18         4.631       19.15       8         4.460       19.89       8         4.309       20.60       14         4.143       21.43       12         4.097       21.68       17         3.926       22.63       19         3.824       23.25       10         3.717       23.92       28         3.689       24.11       45         3.542       25.12       15         3.393       26.32       3         3.329       26.76       4         3.289       27.09       9         3.213       27.74       8         3.176       28.08       13         3.145       28.08       13         3.080       28.91       6         2.989       29.88       16         2.998       30.42       6         2.936       30.42       6         2.988       31.16       8         2.844       31.43       11         2.822       31.68       4         2.714       32.97       7	l e e e e e e e e e e e e e e e e e e e		-
4.631       19.15       8         4.460       19.89       8         4.309       20.60       14         4.143       21.43       12         4.097       21.68       17         3.928       22.63       19         3.824       23.25       10         3.717       23.92       28         3.689       24.11       45         3.542       25.12       15         3.383       26.76       4         3.289       27.09       9         3.213       27.74       8         3.176       28.08       13         3.145       28.38       16         3.088       28.91       6         2.999       29.86       16         2.957       30.20       6         2.936       30.42       5         2.888       31.16       8         2.844       31.43       11         2.822       31.68       4         2.714       32.97       7         2.678       34.08       4         2.791       33.53       3         2.628       34.08       4			
4.460       19.89       8         4.309       20.60       14         4.143       21.43       12         4.097       21.68       17         3.926       22.63       19         3.824       23.25       10         3.717       23.92       28         3.689       24.11       45         3.542       25.12       15         3.383       26.32       3         3.290       26.76       4         3.289       27.09       9         3.213       27.74       8         3.176       28.08       13         3.145       28.36       15         3.086       28.91       6         2.957       30.20       6         2.957       30.20       6         2.936       30.42       5         2.868       31.16       8         2.844       31.43       11         2.822       31.68       4         2.714       32.97       7         2.671       33.53       3         2.628       34.08       4         2.799       34.48       9     <			
4.309			<del>-</del>
4.097       21.68       17         3.926       22.63       19         3.824       23.25       10         3.717       23.92       28         3.689       24.11       45         3.542       25.12       15         3.383       26.32       3         3.329       26.76       4         3.289       27.09       9         3.213       27.74       8         3.176       28.08       13         3.145       28.36       15         3.086       28.91       6         2.989       29.86       16         2.936       30.42       6         2.938       31.16       8         2.844       31.16       8         2.844       31.43       11         2.822       31.68       4         2.714       32.97       7         2.671       33.53       3         2.628       34.08       4         2.599       34.48       9         2.542       35.28       3         2.510       36.76       5         2.471       36.33       8 </td <td></td> <td></td> <td></td>			
3.926 22.63 19 3.824 23.25 10 3.717 23.92 28 3.889 24.11 45 3.542 25.12 15 3.383 26.32 3 3.329 26.76 4 3.289 27.09 9 3.213 27.74 8 3.176 28.08 13 3.146 28.38 16 3.086 28.91 6 2.989 29.96 16 2.957 30.20 6 2.936 30.42 6 2.868 31.16 8 2.844 31.43 11 2.822 31.68 4 2.714 32.97 7 2.671 33.53 3 2.628 34.08 4 2.599 34.48 9 2.542 35.28 3 2.510 36.76 6 2.471 36.33 8 2.439 36.82 5 2.417 37.16 6 2.400 37.44 5 2.381 38.09 4 2.305 39.04 5 2.293 39.26 6		21.43	
3.824 23.25 10 3.717 23.92 28 3.889 24.11 45 3.542 25.12 15 3.393 26.32 3 3.329 26.76 4 3.289 27.09 9 3.213 27.74 8 3.176 28.08 13 3.145 28.38 15 3.086 28.91 6 2.989 29.96 16 2.957 30.20 6 2.936 30.42 6 2.868 31.16 9 2.844 31.43 11 2.822 31.88 4 2.714 32.97 7 2.071 33.53 3 2.628 34.08 4 2.599 34.48 9 2.542 35.28 3 2.510 35.76 5 2.471 38.33 8 2.439 36.82 5 2.417 37.16 6 2.400 37.44 5 2.361 38.09 4 2.305 39.04 5 2.293 39.26 6			
3.717 23.92 28 3.689 24.11 45 3.542 25.12 15 3.383 26.32 3 3.329 26.76 4 3.289 27.09 9 3.213 27.74 8 3.176 28.08 13 3.145 28.38 15 3.086 28.91 6 2.989 29.86 16 2.957 30.20 6 2.936 30.42 5 2.868 31.16 8 2.844 31.43 11 2.822 31.68 4 2.714 32.97 7 2.071 33.53 3 2.628 34.08 4 2.714 32.97 7 2.071 33.53 3 2.628 34.08 4 2.599 34.48 9 2.542 35.28 3 2.510 35.76 5 2.471 38.33 8 2.439 36.82 5 2.417 37.16 6 2.400 37.44 5 2.305 39.04 5 2.293 39.26 6			•-
3.642 26.12 15 3.383 26.32 3 3.329 26.76 4 3.289 27.09 9 3.213 27.74 8 3.176 28.08 13 3.146 28.36 16 3.086 28.91 6 2.989 29.86 16 2.957 30.20 6 2.936 30.42 6 2.936 31.16 8 2.844 31.43 11 2.822 31.88 4 2.714 32.97 7 2.671 33.53 3 2.628 34.08 4 2.599 34.48 9 2.542 35.28 3 2.510 35.76 5 2.471 38.33 8 2.439 36.82 5 2.417 37.16 6 2.400 37.44 5 2.305 39.04 5 2.305 39.04 5			
3.383			
3.329 20.76 4 3.289 27.09 9 3.213 27.74 8 3.176 28.08 13 3.146 28.38 15 3.086 28.91 6 2.989 29.86 16 2.957 30.20 6 2.936 30.42 6 2.868 31.16 9 2.844 31.43 11 2.822 31.68 4 2.714 32.97 7 2.671 33.53 3 2.628 34.08 4 2.599 34.48 9 2.542 35.28 3 2.510 35.76 5 2.471 38.33 8 2.439 38.82 5 2.417 37.16 6 2.400 37.44 5 2.305 39.04 5 2.293 39.26 6			• •
3.213 27.74 8 3.175 28.08 13 3.145 28.38 15 3.086 28.91 6 2.989 29.86 16 2.957 30.20 6 2.938 30.42 5 2.868 31.16 8 2.844 31.43 11 2.822 31.68 4 2.714 32.97 7 2.671 33.53 3 2.628 34.08 4 2.599 34.48 9 2.542 35.28 3 2.510 35.76 5 2.471 38.33 8 2.439 36.82 5 2.417 37.16 6 2.400 37.44 5 2.361 38.09 4 2.305 39.04 5 2.293 39.26 6			,
3.176 28.08 13 3.145 28.38 15 3.086 28.91 6 2.989 29.86 16 2.957 30.20 6 2.936 30.42 5 2.868 31.16 8 2.844 31.43 11 2.822 31.88 4 2.714 32.97 7 2.071 33.53 3 2.628 34.08 4 2.599 34.48 9 2.542 35.28 3 2.510 35.76 5 2.471 38.33 8 2.439 36.82 5 2.417 37.16 6 2.400 37.44 5 2.361 38.09 4 2.305 39.04 5 2.293 39.26 6			
3.146 28.38 15 3.086 28.91 8 2.989 29.88 16 2.957 30.20 6 2.938 30.42 5 2.868 31.16 8 2.944 31.43 11 2.822 31.68 4 2.714 32.97 7 2.671 33.53 3 2.628 34.08 4 2.599 34.48 9 2.542 35.28 3 2.510 35.76 5 2.471 38.33 8 2.439 36.82 5 2.417 37.16 6 2.400 37.44 5 2.361 38.09 4 2.305 39.04 5 2.293 39.26 6			
3.086 28.91 6 2.989 29.86 16 2.957 30.20 6 2.936 30.42 5 2.888 31.16 8 2.844 31.43 11 2.822 31.68 4 2.714 32.97 7 2.671 33.53 3 2.628 34.08 4 2.599 34.48 9 2.542 35.28 3 2.510 35.76 5 2.471 38.33 8 2.439 36.82 5 2.417 37.16 6 2.400 37.44 5 2.361 38.09 4 2.305 39.04 5 2.293 39.26 6			
2.957     30.20     6       2.936     30.42     5       2.868     31.16     8       2.844     31.43     11       2.822     31.68     4       2.714     32.97     7       2.671     33.53     3       2.628     34.08     4       2.599     34.48     9       2.642     35.28     3       2.510     35.76     5       2.471     36.33     8       2.439     36.82     5       2.417     37.16     6       2.400     37.44     5       2.361     38.09     4       2.305     39.04     5       2.293     39.26     6	_		8
2.938     30.42     6       2.868     31.16     8       2.844     31.43     11       2.822     31.68     4       2.714     32.97     7       2.671     33.53     3       2.628     34.08     4       2.599     34.48     9       2.642     35.28     3       2.510     35.76     5       2.471     36.33     8       2.439     36.82     5       2.417     37.16     6       2.400     37.44     5       2.361     38.09     4       2.305     39.04     5       2.293     39.26     6	•		
2.868 31.16 8 2.844 31.43 11 2.822 31.68 4 2.714 32.97 7 2.671 33.53 3 2.628 34.08 4 2.599 34.48 9 2.642 35.28 3 2.510 35.76 5 2.471 38.33 8 2.439 36.82 5 2.417 37.16 6 2.400 37.44 5 2.361 38.09 4 2.305 39.04 5 2.293 39.26 6			_
2.822 31.68 4 2.714 32.97 7 2.671 33.53 3 2.628 34.08 4 2.599 34.48 9 2.642 35.28 3 2.510 36.76 6 2.471 36.33 8 2.439 36.82 5 2.417 37.16 6 2.400 37.44 5 2.381 38.09 4 2.305 39.04 5 2.293 39.26 6	2.868		
2.714     32.97     7       2.671     33.53     3       2.628     34.08     4       2.599     34.48     9       2.542     35.28     3       2.510     35.76     5       2.471     38.33     8       2.439     38.82     5       2.417     37.16     6       2.400     37.44     5       2.361     38.09     4       2.305     39.04     5       2.293     39.26     6			
2.671     33.53     3       2.528     34.08     4       2.599     34.48     9       2.542     35.28     3       2.510     35.76     5       2.471     36.33     8       2.439     36.82     5       2.417     37.16     6       2.400     37.44     5       2.361     38.09     4       2.305     39.04     5       2.293     39.26     6			·-
2.628     34.08     4       2.599     34.48     9       2.642     35.28     3       2.510     35.76     5       2.471     38.33     8       2.439     38.82     5       2.417     37.16     6       2.400     37.44     5       2.361     38.09     4       2.305     39.04     5       2.293     39.26     6			
2.642 35.28 3 2.510 35.76 5 2.471 38.33 8 2.439 36.82 5 2.417 37.16 6 2.400 37.44 5 2.381 38.09 4 2.305 39.04 5 2.293 39.26 6	2.628	34.08	4
2.510 36.76 5 2.471 38.33 8 2.439 36.82 5 2.417 37.16 6 2.400 37.44 5 2.361 38.09 4 2.305 39.04 5 2.293 39.26 6			-
2.471     38.33     8       2.439     38.82     5       2.417     37.16     6       2.400     37.44     5       2.381     38.09     4       2.305     39.04     5       2.293     39.26     6			
2.439     38.82     5       2.417     37.18     6       2.400     37.44     5       2.381     38.09     4       2.305     39.04     5       2.293     39.26     6			8
2.400 37.44 5 2.381 38.09 4 2.305 39.04 5 2.293 39.26 6	2.439		
2.361 38.09 4 2.305 39.04 5 2.293 39.26 6			
2.305 39.04 5 2.293 39.26 6			
· · ·	2.305	39.04	5
2.208 39.90 2	i e		
	2.208	39.90	. 2

POWDER PATTERN (DIFFRACTOGRAMM) Figure 3

7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid -tosylate



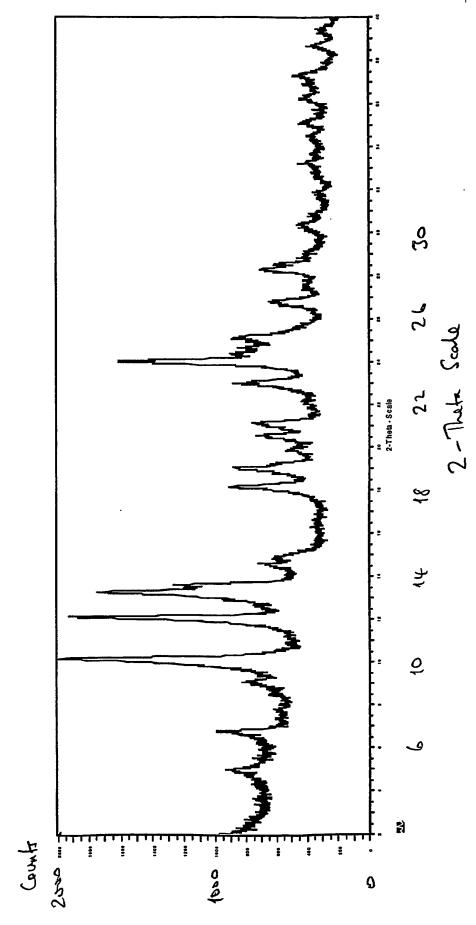
# POWDER PATTERN (D-I-LIST) 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]3-vinyl-3-cephem-4-carboxylic acid hydrogensulfate

Table 4

•		
d value	Angle	Intensity
(Angatrom)	("CuKox)	(Rel. Int.)
18.863	4.68	12
17.700	4.99	21
13.037	6.78	28
9.793	9.02	20
8.751	10.10	100
7.289	12.13	98
6.681	13.24	88
6.515	13.58	56
8.089	14.54	24
5.939	14.90	19
4.887	18.14	39
4.652	19.06	36
4.480	19.80	14
4.321	20.54	24
4.214	21.06	26
4.017	22.11	8
3.868	22.97	35
3.697	24.05	82 27
3.847	24.39	37
3.802	24.70	32
3.533 3.325	25.19 28.70	36 34
3.230 3.230	26.79 27.50	21 7
3.230 3.151	27.59 28.30	, 26
3.121	28.58	20
3.085	28.92	9
2.944	30.33	11
2.901	30.79	7
2.803	31.90	6
2.729	32.80	8
2.692	33.26	11
2.656	33.72	9
2.594	34.55	. 10
2.554	35.11	13
2.476	36.25	14
2.433	36.92	14
2.407	37.32	17
2.321	38.77	11
2.279	39.51	9

POWDER PATTERN (DIFFRACTOGRAMM) Figure 4

7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid -hydrogensulfate



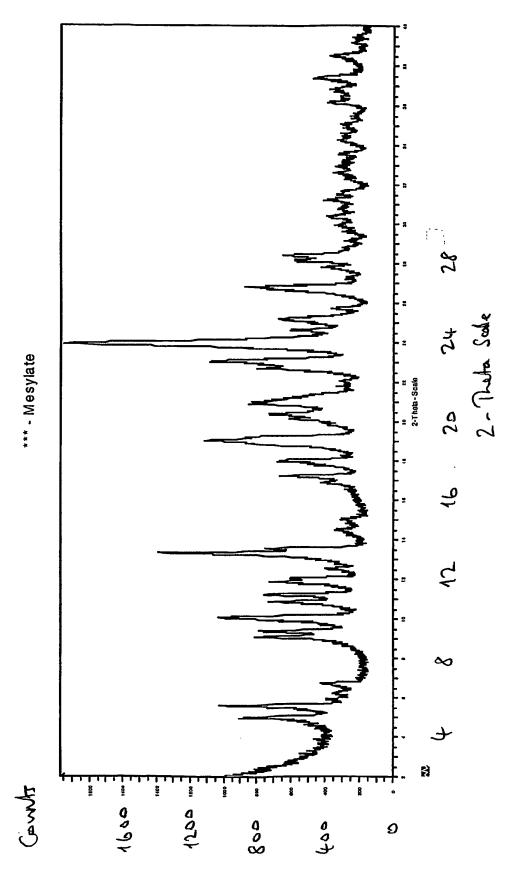
### **POWDER PATTERN (D-I-LIST)** 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid mesylate

Table 5

MESYLATE		***
d value (ModegnA)	Angle (* Cu Ka)	Intensity (Rel. Int.)
17.809	4.98	32
15.689 15.013	5.84 5.88	42 7
14.267	6.19	7
13.158	8.71	12
9.726 9.408	9.09 9.39	36 35
8,804 8,155	10.04	47
7.868	10.84 11.24	31 33
7.459	11.88	31
7,344 7,074	12.04 12.50	24 13
6.650	13.30	69
6.515 6.102	13.58 14.51	32 10
5.878	15.08	8
5.259 5.158	18.85 17.18	14 28
4.979	17.80	21
4.937 4.667	17.95 19.00	28 53
4.815	19.22	38
4.469 4.405	19.89 20.14	13 23
4.359	20.14	32
4.233 4.160	20.97	37
4.022	21.34 22.08	20 9
3.920	22.67	36
3.853 3.718	23.07 23.93	50 100
3.815	24.81	24
3.558 3.528	25.01 25.22	20 28
3.462	25.71	11
3.325 3.266	28.79 27.29	37 7
3.208	27.81	15
3.161 3.134	28.21 28.48	23 27
3.053	29.23	- 6
3,010 2,983	29.66 29.93	7 10
2.938	30.40	13
2.904 2.862	30.77	9
2.829	31.23 31.60	13 9
2.766	32.34	7
2.744 2.718	32.61 32.95	10 10
2.663	33.63	8
2.819 2.589	34.21 34.81	8 8
2.554	35.11	8
2.524 2.478	35.54 36.22	5 13
2.464	38.43	8
2.438 2.403	36.83 37.39	12 17
2.379	37.78	4
2.337 2.301	38.50 39.11	11 4
2.281	39.48	3
L		

POWDER PATTERN (DIFFRACTOGRAMM) Figure 5

7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid -mesylate



# POWDER PATTERN (D-I-LIST) 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]3-vinyl-3-cephem-4-carboxylic acid sulfate

Table 6

SULFATE		· · · · · · · · · · · · · · · · · · ·
d value	Angle	Intensity
(Angstrom)	(°Cu Kox)	(Rel. Int.)
17.924	4.93	15
13.099	6.74	20
9.795	9.02	12
8.806	10.04	48
7.346	12.04	63
6.667	13.27	72
6.548	13.52	51
6.103	14.50	21
5.953	14.87	19
5.377 4.888	16.47 18.13	6 39
4.661	18.13	39 46
4.487	19.86	40 19
4.328	20.51	21
4.203	21.12	32
3.859	23.03	44
3,698	24.05	100
3.616	24.60	32
3.542	25.12	35
3.467	25.68	13
3.333	26.72	21
3.245	27.48	11
3.158	28.23	33
3.125	28.54	21
3.070	29.06	12
2.954	30.23	10
2.907	30.73	7
2.844	31.43	3
2.814	31.77	5
2.722	32.88	8
2.695	33.22	10
2.859 2.803	33.68	7
2.503 2.561	34.42 25.04	9 11
2.561 2.483	35.01 38.15	11 10
2.435 2.435	36.15 36.88	10
2.408	37.32	14
2.327	38.67	9
2.288	39.35	5
		_

POWDER PATTERN (DIFFRACTOGRAMM) Figure 6

7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid -sulfate

